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(56) Documents cited

GB 1512766 A WO 83/03796 A1 US 4399175 A US 4212925 A US 3055831 A US 2808338 A

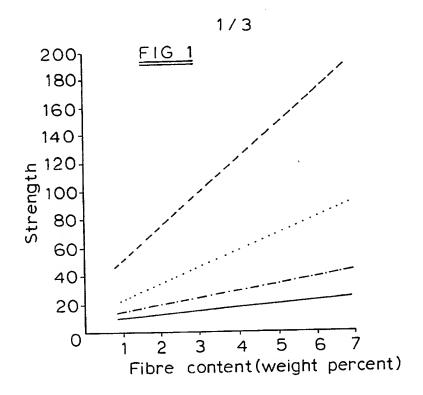
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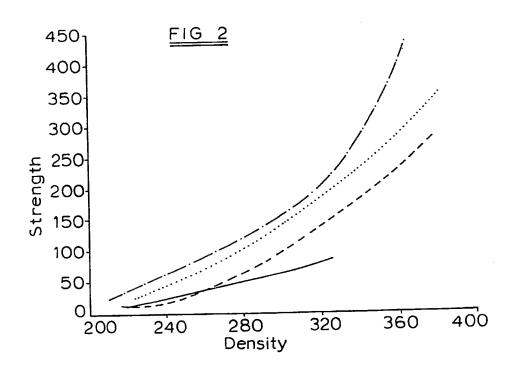
(54) Thermal insulation material

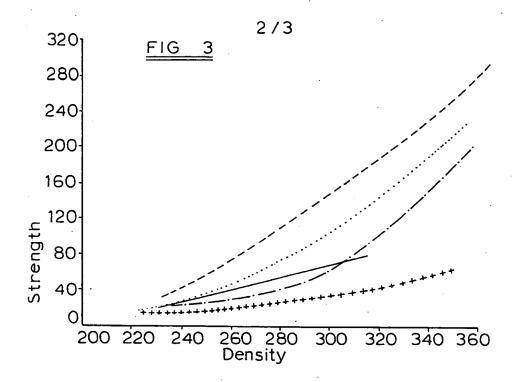
(57) A microporous thermal insulation material comprises an intimate mixture of dry particulate microporous material and reinforcing glass fibres. The glass constituting the glass fibres contains not more than 1 per cent by weight Na₂O and preferably has the following composition:

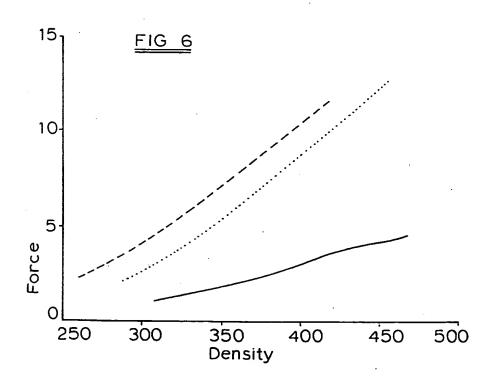
SiO₂	50 - 100 per cent by weight
Al ₂ O ₃	up to 25 per cent by weight
B ₂ O ₃	up to 8 per cent by weight
MgO	up to 10 per cent by weight
CaO	up to 21 per cent by weight
Na₂O	up to 1 per cent by weight
K₂O	up to 2 per cent by weight
Fe ₂ O ₃	up to 1 per cent by weight
F ₂	up to 1 per cent by weight.

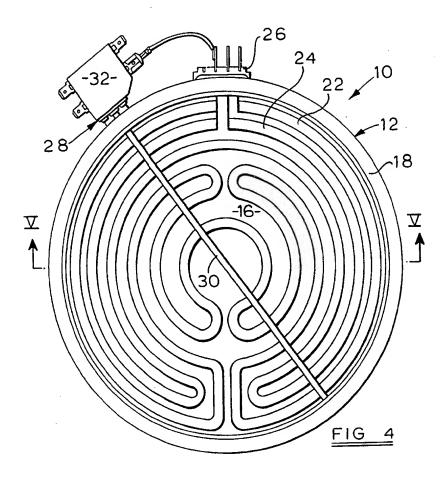
The material may be based on pyrogenic silica and titanium dioxide.

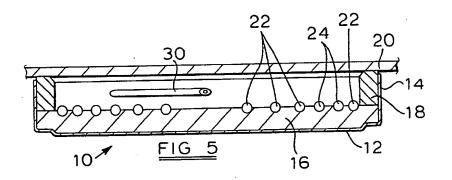












MICROPOROUS THERMAL INSULATION MATERIAL

The present invention relates to microporous thermal insulation material and more particularly relates to microporous thermal insulation material containing glass fibre reinforcement.

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The term 'microporous' is used herein to identify porous or cellular materials in which the ultimate size of the cells or voids is less than the mean free path of an air molecule at NTP, i.e. of the order of 100 nm or smaller. A material which is microporous in this sense will exhibit very low transfer of heat by air conduction (that is collisions between air molecules). Such microporous materials include aerogel, which is a gel in which the liquid phase has been replaced by a gaseous phase in such a way as to avoid the shrinkage which would occur if the gel were dried directly from a liquid. A substantially identical structure can be obtained by controlled precipitation from solution, the temperature and pH being controlled during precipitation to obtain an open lattice precipitate. Other equivalent open lattice structures include pyrogenic (fumed) and electrothermal types in which a substantial proportion of the particles have an ultimate particle size less than 100 nm. Any of these materials, based for example on silica, alumina or other metal oxides, may be used to prepare a composition which is microporous as defined above.

Microporous thermal insulation is described, for example, in US-A-2 808 338 as comprising a reinforcing skeleton of fine staple reinforcing fibres which may be either organic or inorganic, a substantial amount, and preferably at least 45 per cent by weight, of a particulate filler material having a porous or fibrillate structure such as silica aerogel and, preferably, a substantial amount of finely divided opacifier materials. Suitable reinforcing fibres are said to include various types of asbestos fibres of reinforcing grade, cleaned mineral fibres, fine diameter glass fibres, preferably pre-treated, as with acid, to roughen the surface or otherwise to improve the surface adhesion characteristics, and organic fibres. A similar range of reinforcing fibres is disclosed in US-A-3 055 831.

US-A-4 212 925 describes an insulation material that is microporous in that it contains pyrogenic silica, an opacifier and an inorganic reinforcing fibre. The inorganic reinforcing fibre may be glass wool, rock wool, slag wool or ceramic fibres such as those which are obtained by melting aluminium oxide and silicon dioxide. US-A-4 399 175 describes a similar microporous thermal insulation material which may contain reinforcing fibres such as aluminium silicate fibres, quartz or glass fibres, or ceramic fibres.

US-A-4 221 672 describes the use of glass fibres in some detail. According to this reference it is conventional to

employ aluminosilicate reinforcing fibres, but that the use of alumina fibres increases the tolerance of the microporous thermal insulation material to heat. On the other hand, glass fibres or mineral wool fibres resulted in high shrinkage occurring at lower temperatures, of the order of $700~^{\circ}\text{C}$.

The prior art shows that although the use of glass reinforcing fibres as such has been proposed, the maximum temperature of use of microporous thermal insulation material containing such fibres is limited to some 700 °C due to excessive shrinkage of the insulation material at such temperatures.

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- It is an object of the present invention to provide a microporous thermal insulation material containing glass fibre reinforcement which has a maximum temperature of use in excess of 700 $^{\circ}$ C.
- According to the present invention there is provided a microporous thermal insulation material comprising an intimate mixture of a dry particulate microporous material and reinforcing glass fibres, wherein the glass constituting the glass fibres contains not more than 1 per cent by weight Na₂O.

Such glass fibres are available commercially as E-glass fibres.

The glass may have the following composition:

	SiO2	50 - 100 per cent by weight
	A7203	up to 25 per cent by weight
	B ₂ O ₃	up to 8 per cent by weight
5	MgO	up to 10 per cent by weight
	Ca0	up to 21 per cent by weight
	Na ₂ O	up to 1 per cent by weight
	K ₂ O	up to 2 per cent by weight
	Fe ₂ O ₃	up to 1 per cent by weight
10	F ₂	up to 1 per cent by weight.

The glass may have substantially the following composition:

	sio	54 to 55 per cent by weight
	A1203	14 to 15 per cent by weight
15	B ₂ O ₃	7 to 8 per cent by weight
	MgO	0.3 to 3 per cent by weight
	CaO	18 to 21 per cent by weight
	Na ₂ O	up to 0.6 per cent by weight
	K ₂ O	up to 0.2 per cent by weight
20	Fe ₂ O ₃	0.2 to 0.4 per cent by weight
	F, .	up to 1 per cent by weight.

Alternatively, the glass may have substantially the following composition:

25	SiO2	60 per cent by weight
	A12O3	25 per cent by weight
	MgO	6 per cent by weight
	CaO	9 per cent by weight

The glass fibres may be chopped from continuous glass fibres. Such fibres are readily available from a number of manufacturers. Chopped strand glass is made by melting a suitable glass formulation in a tank from which it flows through small diameter bushings and is then drawn into continuous filaments, for example 5 to 24 microns diameter, and these are dressed with an organic size and gathered to form strands. These strands are then chopped to discrete lengths. In contrast to other mineral fibres, chopped strand glass is substantially free from any non-fibrous material such as shot and is of substantially uniform diameter.

The glass fibres may have a length from 4 to 50 mm, and preferably may have a length from 6 to 25 mm.

The glass fibres may have a diameter in the range from 3 to 20 microns, and preferably may have a diameter in the range from 6 to 15 microns.

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The microporous thermal insulation material may contain from 1 to 20 per cent by weight of glass fibres, and preferably may contain from 3 to 15 per cent by weight of glass fibres.

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For a better understanding of the present invention and to show more clearly how it may be carried into effect reference will now be made, by way of example, to the accompanying drawings in which:

Figure 1 is a graph illustrating the variation of the flexural strength of microporous thermal insulation material with fibre content for different fibre types;

Figure 2 is a graph illustrating the variation of the flexural strength of microporous thermal insulation material with density for different fibre types;

Figure 3 is a graph illustrating the variation of the flexural strength of microporous thermal insulation material with density for different fibre contents;

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Figure 4 is a plan view of a radiant heater incorporating a peripheral wall made of microporous thermal insulation material according to the present invention;

Figure 5 is a cross-sectional view taken along the line V-V in Figure 4; and

Figure 6 is a graph illustrating the variation of the strength of rings of microporous thermal insulation material with density for different mixtures.

The invention will be explained with reference to the following Examples.

EXAMPLE 1 (COMPARATIVE)

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A block of microporous thermal insulation material was made by mixing together in a blade-type mixer a mixture of 60 per cent by weight of pyrogenic silica available from Cabot Corporation under Trade Mark CABOSIL M5, the silica having a nominal specific surface area of 250 m²/g as measured by the B.E.T method, 33.3 per cent by weight of a particulate opacifier in the form of titanium dioxide and 6.7 per cent by weight of aluminosilicate (or ceramic) fibres available from The Carborundum Company Limited under the Trade Mark FIBERFRAX. The materials were mixed together in order to obtain a homogeneous mixture.

The mixture was compacted into a cylindrical block having a diameter of 110 mm and a thickness of 25 mm, the block having a density of 320 kg/m³ and was heated at a temperature of 800°C for a period of 24 hours. When the block had cooled it was established that the block had shrunk by 0.35 per cent in the diametral direction and 0.80 per cent in the axial direction. Such a material is fully suitable for use as a thermal insulation material at a temperature of 800 °C.

25 EXAMPLE 2 (COMPARATIVE)

A block of microporous thermal insulation material was made by mixing together a mixture of 60 per cent by weight of pyrogenic silica available from Cabot Corporation under Trade Mark CABOSIL M5, 33.3 per cent by weight of a particulate opacifier in the form of titanium dioxide and 6.7 per cent by weight of chopped glass strand available from Glaswerk Schüller GmbH under the Trade Mark MICROLITH, the fibres having a nominal length of 25 mm and a nominal diameter of 12 microns, the glass having the following composition:

10	SiO2	65 per cent by weight
	A1 ₂ O ₃	4 per cent by weight
	B ₂ O ₃	5 per cent by weight
	MgO	3 per cent by weight
	CaO	14 per cent by weight
15	Na ₂ O	8.5 per cent by weight
	Fe ₂ O ₃	0.3 per cent by weight

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together with incidental ingredients and impurities.

The mixture was compacted into a cylindrical block having substantially the same dimensions as in Example 1, the block having a density of 319 kg/m 3 , and was heated at a temperature of 800 $^{\circ}$ C for a period of 24 hours. When the block had cooled it was established that the block had shrunk by 39.5 per cent in the diametral direction and 18.5 per cent in the axial direction. Such a material is clearly unsuitable for use as a thermal insulation material at a temperature of 800 $^{\circ}$ C.

pyrogenic silica available from Cabot Corporation under Trade Mark CABOSIL M5, 33.3 per cent by weight of a particulate opacifier in the form of titanium dioxide and 6.7 per cent by weight of chopped glass strand available from Glaswerk Schüller GmbH under the Trade Mark MICROLITH, the fibres having a nominal length of 25 mm and a nominal diameter of 12 microns, the glass having the following composition:

10	SiO ₂	65 per cent by weight
	A12O3	4 per cent by weight
	B ₂ O ₃	5 per cent by weight
	MgO	3 per cent by weight
	CaO	14 per cent by weight
15	Na ₂ O	8.5 per cent by weight
	Fe ₂ O ₃	0.3 per cent by weight

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together with incidental ingredients and impurities.

The mixture was compacted into a cylindrical block having substantially the same dimensions as in Example 1, the block having a density of 319 kg/m 3 , and was heated at a temperature of 800 0 C for a period of 24 hours. When the block had cooled it was established that the block had shrunk by 39.5 per cent in the diametral direction and 18.5 per cent in the axial direction. Such a material is clearly unsuitable for use as a thermal insulation material at a temperature of 800 0 C.

 800^{0} C for a period of 24 hours. When the block had cooled it was established that the block had shrunk by 1.54 per cent in the diametral direction and 1.51 per cent in the axial direction. Such a material is generally suitable for use as a thermal insulation material at a hot face temperature of $800~^{0}$ C.

EXAMPLE 4

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10 Example 3 was repeated but substituting in the block of microporous thermal insulation material chopped E-glass strand available from Vetrotex International under the Trade Mark VETROTEX EB107DC/5EC14300 12 mm (P312 SP219) in place of the chopped E-glass strand from Owens Corning Fiberglas Corporation, the fibres having a nominal length of 12 mm and a nominal diameter of 14 microns.

The mixture was compacted into a cylindrical block having similar dimensions to those in Example 1, the block having a density of 313 kg/m³, and was heated at a temperature of 800° C for a period of 24 hours. When the block had cooled it was established that the block had shrunk by 1.74 per cent in the diametral direction and 1.20 per cent in the axial direction. Such a material is generally suitable for use as a thermal insulation material at a hot face temperature of 800° C.

EXAMPLE 5

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A block of microporous thermal insulation material was made by mixing together, in the same manner as Example 2, a mixture of 60 per cent by weight of pyrogenic silica available from Cabot Corporation under Trade Mark CABOSIL M5, 33.3 per cent by weight of a particulate opacifier in the form of titanium dioxide and 6.7 per cent by weight of chopped R-glass strand available from Vetrotex International under the Trade Mark VETROTEX Rc10 4.5mm p388 code CR98D, the fibres having a nominal length of 4.5 mm and a nominal diameter of 10 microns, the glass having substantially the following composition:

15	SiO2	60 per cent by weight
	A1203	25 per cent by weight
	MgO	6 per cent by weight
	CaO	9 per cent by weight

The mixture was compacted into a cylindrical block having similar dimensions to those in Example 1, the block having a density of 320 kg/m³, and was heated at a temperature of 800°C for a period of 24 hours. When the block had cooled it was established that the block had shrunk by 0.83 per cent in the diametral direction and 0.80 per cent in the axial direction. Such a material is generally suitable for use as a thermal insulation material at a hot face temperature of 800 °C.

EXAMPLE 6

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Tests were carried out to determine the effect of fibre content in the mixture. Blocks of microporous thermal insulation material were made by mixing together and compacting, generally in the same manner as Example 2, a mixture of 60 per cent by weight of pyrogenic silica available from Cabot Corporation under the Trade Mark CABOSIL M5, and 40 per cent by weight of a mixture of particulate opacifier in the form of titanium dioxide and chopped glass strand available from Vetrotex International under the Trade Mark VETROTEX, the fibres having a nominal length of 12 mm and a nominal diameter of 12 microns and a glass composition substantially the same as that in Example 3 above. Glass fibres were incorporated into the mix from which the blocks were manufactured in the proportions by weight of 5 per cent, 10 per cent, 15 per cent and 20 per cent.

20 The blocks were heated at a temperature of 800°C for a period of 24 hours. When the blocks had cooled the following shrinkage figures were obtained:

Fibre content	Diametral Shrinkage	Axial Shrinkage
5 per cent	0.91 %	0.84 %
10 per cent	1.11 %	0.93 %
15 per cent	1.22 %	0.76 %
20 per cent	1.41 %	0.67 %

Table 1

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By way of comparison, a block containing 5 per cent by weight of FIBERFRAX fibres made and heated in the same manner was found to have a diametral shrinkage of 0.28 per cent and an axial shrinkage of 0.17 per cent.

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EXAMPLE 7

As described hereinabove, microporous thermal insulation material is noted for its particularly low conductivity. Clearly, any new form of microporous thermal exhibit similarly low insulation should conductivity. We have manufactured blocks of microporous thermal insulation material in accordance with the method outlined in Example 5, but using a pyrogenic silica available from Cabot Corporation under the Trade Mark CABOSIL MS55, in place of M5 silica, in combination with Fibre Types 3, 4 and 5 from Example 4. The material was compacted to a density of 320 kg/m³. We have compared the thermal conductivity of blocks containing glass fibres with the thermal conductivity of equivalent blocks containing FIBERFRAX fibres and the results are summarised in Table 2 below:

	Fibre content (per cent by weight)	Thermal (W/	conductivity (m K))
		200 °C mean	400 °C mean
5	Fibre Type 3 3.0 5.0 6.7	0.0254 0.0261 0.0279	0.0323 0.0340 0.0377
10	Fibre Type 4 3.0 5.0 6.7	0.0252 0.0256 0.0271	0.0314 0.0319 0.0344
15	Fibre Type 5 3.0 5.0 6.7	0.0251 0.0258 0.0275	0.0320 0.0337 0.0360
20	FIBERFRAX (Comparative) 3.0 5.0 6.7	0.0242 0.0244 0.0251	0.0303 0.0304 0.0308

Table 2

25 We have also measured the thermal conductivity of flat panels of microporous thermal insulation material compacted to 240 kg/m³ within a porous envelope of glass fibre material using CABOSIL MS55 grade silica from Cabot Corporation, titanium dioxide and either Fibre Type 4 or 30 FIBERFRAX. The results are summarised in Table 3 below:

Fibre content (per cent by weight)	Thermal conductivity (W/ (m K))	
	200 °C mean	400 °C mean
Fibre Type 4 3.0 5.0 6.7	0.0246 0.0250 0.0254	0.0301 0.0316 0.0322
FIBERFRAX (Comparative) 6.7	0.0240	0.0303

Table 3

Thus, while the thermal conductivity of blocks of the insulation material containing Fibre Types 3, 4 and 5 falls with decreasing fibre content, it never quite falls to the thermal conductivity of blocks containing equivalent amounts of FIBERFRAX. On the other hand, the thermal conductivity of panels containing 3 per cent by weight of Fibre Type 4 is comparable with the thermal conductivity of equivalent panels containing 6.7 per cent by weight of FIBERFRAX. We believe this is because the glass fibres in the panels are more orientated than the FIBERFRAX fibres.

EXAMPLE 8

Microporous thermal insulation material, being made from compacted particulate materials, is a relatively weak and friable material. One consideration when manufacturing new forms of microporous thermal insulation material is therefore the strength of the material and the degree to which it can be handled. We have carried out tests on the

flexural strength of microporous thermal insulation materials containing glass fibres.

Test procedure

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A rectangular slab of the microporous thermal insulation material is placed in a standard three-point flexural strength test rig ensuring that the slab is centralised between the three loading bars. The load is then increased until the slab fails and the maximum force in kN is noted. The flexural strength of the slab in kN/ m^2 can be determined from the maximum force supported by the slab and the dimensions of the slab in accordance with the following equation:

Flexural Strength -
$$\frac{3 \times P \times s}{2 \times a^2 \times b}$$

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where:

P = maximum force (kN) supported by the panel

s = support span (m) between loading bars

a = thickness (m) of the panel

b = width (m) of the panel.

A first test was carried out on blocks made from a number of different microporous thermal insulation mixtures generally in accordance with Example 7 and based on Fibre Types 4 and FIBERFRAX with the fibre content of the mixture varying between 1 and 6.7 per cent by weight and the

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mixture compacted to average densities of 240 kg/m³ and 320 kg/m³, although it should be noted that blocks containing FIBERFRAX could not be made successfully below 2 per cent by weight of fibre. The results are summarised in Figure 1 which is a graph showing the variation in flexural strength of the blocks (measured in kN/m^2) with fibre In Figure 1, the dashed line represents the content. flexural strength of material containing Fibre Type 4 and compacted to a density of 320 kg/m^3 , the dotted line represents comparative data on the strength of material containing FIBERFRAX and compacted to a density of 320 kg/m³, the dot-dash line represents the strength of material containing Fibre Type 4 and compacted to a density of 240 kg/m³ and the full line represents comparative data on the strength of material containing FIBERFRAX compacted to a density of 240 kg/m³. Figure 1 shows that the flexural strength of microporous thermal insulation material in general increases with increasing fibre content and that the flexural strength of microporous thermal insulation material containing glass fibres is, in general, superior to the flexural strength of microporous thermal insulation material containing FIBERFRAX.

A second test was carried out on slabs moulded from a number of different microporous thermal insulation mixtures generally in accordance with Example 6 and based on Fibre Types 3, 4 and 5 and FIBERFRAX each having a fibre content of 6.7 per cent by weight and the mixture compacted to

densities in the range from 200 to 400 kg/m 3 . The results are summarised in Figure 2 which is a graph showing the variation in flexural strength of the slabs (measured in kN/m^2) with material density (measured in kg/m^3). In Figure 2, the dashed line represents the flexural strength of material containing Fibre Type 3, the dotted represents the strength of material containing Fibre Type 4, the dot-dash line represents the strength of material containing Fibre Type 5 and the full line represents comparative data on material containing FIBERFRAX. 2 shows not only that microporous thermal insulation material containing glass fibres generally possesses a flexural strength higher than material containing FIBERFRAX, but that the strength also depends on the type of glass fibres used. The glass fibres of Fibre Type 3 have a length of 6 mm, those of Fibre Type 4 have a length of 12 mm, and those of Fibre Type 5 have a length of 25 mm. Thus, within the range of the tests we have carried out, it can be seen that flexural strength increasing fibre length.

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A third test was carried out on slabs moulded from a number of different microporous thermal insulation mixtures based solely on Fibre Type 4 having fibre contents of 1 per cent by weight, 3 per cent by weight, 5 per cent by weight and 6.7 per cent by weight and the mixture being compacted to densities in the range from 200 to 360 kg/m³. The results are summarised, and compared with the results of the

mixture containing FIBERFRAX from the second test, in Figure 3 which is a graph showing the variation in flexural strength of the panels (measured in kN/m^2) with the material density (measured in kg/m^3). In Figure 3 the dashed line represents the flexural strength of material containing 6.7 per cent by weight glass fibre, the dotted line represents the strength of material containing 5 per cent by weight glass fibre, the dot-dash line represents the strength of material containing 3 per cent by weight glass fibre, the line of "+" characters represents the strength of material containing only 1 per cent by weight of glass fibre, and the full line represents the strength of material containing 6.7 per cent by weight of FIBERFRAX. shows that the flexural strength of microporous thermal insulation material increases with an increasing proportion of glass fibres, but more significantly shows that the of microporous thermal insulation containing glass fibres is considerably stronger than material containing a similar proportion of FIBERFRAX.

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The microporous thermal insulation material according to the present invention can be used, for example, in the manufacture of panels enclosed within confining envelopes and in the manufacture of moulded shapes. The material is resistant to shrinkage up to at least 800 °C, and possibly higher, and chopped glass strand, in contrast to ceramic fibres, is not respirable. One specific use of the microporous thermal insulation material according to the

present invention will now be described with reference to Figures 4 and 5.

The radiant electric heater 10 shown in Figures 4 and 5 comprises a container in the form of a metal dish 12 with an upstanding rim 14 and containing a layer of electrical and thermal insulating material 16, such as a microporous thermal insulation material. A ring-shaped insulating wall 18 of microporous thermal insulation according to the present invention extends around the inside of the rim 14 of the dish 12, on top of the layer 16 and protruding slightly above the edge of the rim 14. When installed in a glass ceramic top cooker the wall 18 is pressed against the underside of a glass ceramic cooking surface 20, the heater 10 being held in position by a spring or other mounting device (not shown).

The layer 16 supports two coiled bare resistance-wire heating elements 22 and 24 arranged in multiple concentric, generally circular portions within and adjacent the wall 18. The coiled elements 22 and 24 are secured to the layer 16 by, for example, staples held by friction in the insulating material of the layer 16, or by gluing to the layer 16 or to stakes inserted therein. The ends of the heating elements 22 and 24 are coupled to respective conductors in an electrical conductor block 26 mounted at the edge of the dish 12.

As is customary with radiant heaters for glass ceramic top cookers, a temperature sensitive rod limiter 28 is provided with its probe 30 extending across the heater 10. This probe typically comprises a fused silica tube containing a metal rod, which is preferably plated with a reflective material, such as silver, as described in GB-A-2 146 431. A snap-action switch 32 controlled by the probe 30 is provided for connection in series with the heating elements 22 and 24, to prevent heating of the cooktop 20 above its maximum safe temperature. The limiter switch 32 is connected to the ends of the two heating elements 22 and 24.

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Clearly the configuration of the heater can be varied. For example the overall shape of the heater can be changed as can the number and type of heating elements, possibly to include infra-red lamps. The important aspect of the radiant heater shown in Figures 4 and 5 is that the wall 18 microporous thermal insulation material is made according to the present invention. It is usual to make the wall 18 from a tube made by vacuum forming ceramic fibre material which is hardened with the aid of a binder such as silica sol and an organic starch. However, cutting the tube into individual rings and trimming the rings to fit the heater 10 gives rise to considerable quantities of dust which is undesirable, and the organic starch requires to be burnt out in order to avoid smoke and smell from the Moreover, such rings are first use of the heater.

generally too weak to use without re-hardening with silica sol. The problem has always been to find an alternative inorganic insulating material for the peripheral wall 18 that is not only free from dust, but which also has sufficient mechanical strength to withstand the stresses incurred during manufacture of the heater and subsequent assembly of the heater into a cooking appliance.

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Depending on its configuration and any additional hardening treatment a peripheral wall 18 will withstand a diametral force of some 4 to 11 N before failure. We have tested a conventional microporous thermal insulation material comprising 60 per cent by weight of CABOSIL M5 grade silica, 28.5 per cent by weight of titanium dioxide and 11.5 per cent by weight of aluminosilicate fibres in the form of FIBERFRAX by moulding the microporous thermal insulation material into rings at a range of densities from 310 to 460 kg/m 3 . Only at densities of nearly 450 kg/m 3 did the rings withstand a force in excess of 4 N. We have also tested microporous thermal insulation material containing glass fibres according to the present invention by moulding the microporous thermal insulation material into rings. A first mixture comprised 60 per cent by weight of CABOSIL M5 grade silica, 30 per cent by weight of titanium dioxide and 10 per cent by weight of Fibre Type 3, and a second mixture comprised 60 per cent by weight of CABOSIL M5 grade silica, 25 per cent by weight of titanium dioxide and 15 per cent by weight of Fibre Type 3. The results are summarised in Figure 6 in which the mixture containing 15 per cent by weight of Fibre Type 3 is shown as a dashed line, the mixture containing 10 per cent by weight of Fibre Type 3 is shown as a dotted line, and the mixture containing 11.5 per cent by weight of FIBERFRAX is shown as a full line. As can be seen from Figure 6, the mixture with 15 per cent by weight of glass fibres withstands a force of 4 N at a density of less than 300 kg/m³ and a force of 11 N at a density of about 410 kg/m³, while even the mixture with 10 per cent by weight of glass fibres withstands a force of 4 N at a density of about 330 kg/m³ and a force of 11 N at a density of less than 440 kg/m³.

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We believe this clearly demonstrates the considerable strength advantages of the microporous thermal insulation material according to the present invention, which advantages are coupled with the unexpected resistance of the material to shrinkage at temperatures even up to 800 °C.

CLAIMS

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- 1. A microporous thermal insulation material comprising an intimate mixture of a dry particulate microporous material and reinforcing glass fibres, wherein the glass constituting the glass fibres contains not more than 1 per cent by weight Na_2O .
- A microporous thermal insulation material as claimed
 in claim 1, wherein the glass has the following composition:

:	SiO2	50 - 100 per cent by weight
	A1203	up to 25 per cent by weight
	B ₂ O ₃	up to 8 per cent by weight
15	MgO	up to 10 per cent by weight
	CaO	up to 21 per cent by weight
	Na ₂ O	up to 1 per cent by weight
	κ ₂ ο	up to 2 per cent by weight
	Fe ₂ O ₃	up to 1 per cent by weight
20	F,	up to 1 per cent by weight.

3. A microporous thermal insulation material as claimed in claim 2, wherein the glass has substantially the following composition:

25 SiO₂ 54 to 55 per cent by weight $Al_2O_3 \qquad 14 \text{ to 15 per cent by weight}$ $B_2O_3 \qquad 7 \text{ to 8 per cent by weight}$ MgO 0.3 to 3 per cent by weight

	CaO	18 to 21 per cent by weight
	Na ₂ O	up to 0.6 per cent by weight
	K₂O	up to 0.2 per cent by weight
	Fe ₂ O ₃	0.2 to 0.4 per cent by weight
5	F ₂	up to 1 per cent by weight.

4. A microporous thermal insulation material as claimed in claim 2, wherein the glass has substantially the following composition:

10	SiO2	60	per	cent	by	weight	
	A1203	25	per	cent	bу	weight	
	MgO	6	per	cent	bу	weight	
	CaO	9	per	cent	bу	weight	

- 15 5. A microporous thermal insulation material as claimed in any preceding claim, wherein the glass fibres are chopped from continuous glass fibres.
- 6. A microporous thermal insulation as claimed in any preceding claim, wherein the glass fibres have a length from 4 to 50 mm.
- A microporous thermal insulation material as claimed in claim 6, wherein the glass fibres have a length from 6
 to 25 mm.

- 8. A microporous thermal insulation material as claimed in any preceding claim, wherein the glass fibres have a diameter in the range from 3 to 20 microns.
- 5 9. A microporous thermal insulation material as claimed in claim 8, wherein the glass fibres have a diameter in the range from 6 to 15 microns.
- 10. A microporous thermal insulation material as claimed 10 in any preceding claim, wherein the material contains from 1 to 20 per cent by weight of glass fibres.
- 11. A microporous thermal insulation material as claimed in claim 10, wherein the material contains from 3 to 15 per15 cent by weight of glass fibres.
 - 12. A microporous thermal insulation material as claimed in claim 1 and substantially as hereinbefore described with reference to Examples 3 to 9.

Patents Act 1977 Examiner's report to the Comptroller under Section 17 (The Search Report)

Application number

9202608.7

Relevant Technical fields	Search Examiner
(i) UK CI (Edition)	
(ii) Int CI (Edition 5) CO4B	MISS M M KELMAN
Databases (see over) (i) UK Patent Office	Date of Search
(ii) ONLINE DATABASE: WPI	23 MARCH 1992
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Documents considered relevant following a search in respect of claims

1 TO 12

Category (see over)	Identity of document and relevant passages	Relevant to claim(s)
Y	GB 1512766 (ACI TECHNICAL) see Claim 1 and page 2, lines 90 to 94	1 to 12
Y	WO 83/03796 A1 (HEXCEL) see page 2, lines 21 to 26, and page 4 lines 29 to 32	1 to 12
Y	US 4399175 A (KUMMER MEHR) see column 3, lines 51 to 58	1 to 12
Y	US 4212925 A (WACKER-CHEMIE) see column 2	1 to 12
Y	US 3055831 A (BARNETT) see columns 5, 7 and 8	1 to 12
Y .	US 2808338 A (BRUNO) see the claims and column 2	
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Category	Identity of document and relevant passages				
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		1			

Categories of documents

- X: Document indicating lack of novelty or of inventive step.
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